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(54) Title: LIQUID PACKAGING PAPER

(57) Abstract: An improved liquid paperboard product with reduced edgewick sizing properties is produced through the incorporation of a calcium carbonate filler in amounts of up to 10.0 weight percent. The filler enhances the retention and reactivity of a sizing agent such as alkyl ketene dimers which in combination further inhibits liquid penetration into liquid paperboard substrates.

A LIQUID CONTAINER PAPER

Field of the Invention

The present invention relates generally to paper products and more particularly paper containers useful in the packaging of liquids and solutions. More specifically, the present invention relates to improved liquid packaging board used in the production of these liquid containers and processes for its preparation.

Background of the Invention

Edgewick sizing is the resistance of liquid penetration in liquid packaging board. The resistance to liquid penetration along the exposed edge inside a paperboard container helps to maintain the strength and rigidity of the container over its life cycle. This type of board has historically been made without filler because of the presumed negative effects to the boards strength and especially it's sizing properties.

Considerable amounts of time and money are spent each year in the development of designs and attractive logos on artwork that enhance the presentation of packaged materials to attract the attention of the consumer. The design not only envelopes the size and shape of the package, but just the right combination of colors and graphics go a long way in determining whether the packaged product is a market success or failure.

Liquid materials packaged in paper stock containers such as dairy products, juices and other beverages present added production and storage factors that must be overcome. Liquids such as these as well as oils, greases and other solutions can penetrate the paperboard comprising the package and subsequently stain the exposed surface of the package and ruin the aesthetic appearance of the graphics, colors and design on the outer surface used to attract the consumer as well as destroying the structural integrity of the

container. Obviously, this is not a desirable situation.

Previous efforts that focused on eliminating the problem of stains produced by liquids, oils, grease and beverage penetration into the packaging stock have been directed at preventing the liquid from penetrating into the stock. Foil laminated packaging stock is one means but this is relatively expensive. Other methods of preventing grease penetration into stock which have heretofore been employed are polyolefin coatings, fluorocarbon coatings, saturation with paraffin or microcrystalline waxes, coating one or both sides of the stock with polymer fortified waxes, and the like. However, these methods have proven to be costly and have demonstrated limited success.

Containers for packaging liquid products, particularly diary products such as milk and cream, are made out of coated paper-based board. The coating may be on one side of the board but is generally on both sides and is usually polyethylene, although other water-proofing substances may be used.

To function effectively in such a container, the board must be resistant to the effects of the liquid. For liquid diary products, the most aggressive penetrating component of the liquid is generally lactic acid. The most vulnerable part of the board tends to be its cut edge. Board manufacturers have therefore investigated ways to improve the resistance of board to edge penetration by lactic acid -containing liquids. It is known that a board sized with a ketene dimer (KD) has good resistance to edge penetration by lactic acid-containing liquids.

In many instances, it is undesirable to employ a metallized substrate as a packaging material particularly where exterior coloration and/or printing is desired on the surface of the package and structural integrity is paramount. It may also be impractical or too expensive to employ a barrier to liquid, oil and/or grease absorption. In such a case, it would be desirable to at least provide an easily printable packaging material which could mask the appearance of unsightly oleaginous stains from the eyes of a prospective purchaser should penetration of the paperboard by the liquid occur.

There is still much room for improvement in the edgewick sizing characteristics of paperboard. In the past, in order to maintain a good edgewick sizing, especially with respect to beverages containing lactic acid, i.e. milk, high levels of alkyl ketene dimer (a cellulose reactive sizing agent) and retention aids and thermosetting resins (such as Kymene ® Hercules, Inc. Wilmington, Delaware) were used to promote hard sizing along the edge of the board that is most susceptible to liquid penetration and degradation. Most paperboard used in the production of liquid containers had to be made without filler because of the negative results with respect to paperboard strength and sizing properties.

It is an object of the present invention to improve the edgewick sizing properties of paper board useful in the construction and manufacture of liquid containers and packaging through the incorporation of a filler material. It is a further object of the present invention to prepare a paperboard product for use in liquid containers and packaging wherein the filler comprises up to ten percent (10%) of the paper board with or without the inclusion of a retention aid.

Summary of the Invention

The present invention comprises a paper board product and process for its preparation useful in the construction and manufacture of liquid containers, aseptic packaging and packaging for dairy products, juices, carbonated and uncarbonated soft drinks, hot beverages and other packaged liquids. The paperboards' edgewick sizing properties are improved through the incorporation of a calcium carbonate filler which helps to increase the retention and reactivity of a sizing agent such as alkyl ketene dimer (AKD) or alkenyl succinic anhydride (ASA) which inhibit liquid penetration.

Detailed Description of the Invention

When mineral particles such as calcium carbonate are used as filler in the manufacture and production of liquid paperboard, the internal cohesive properties of the paper is lessened since the mineral particles present larger surface areas than the wood fibers alone. Mineral particle filler would be expected to interfere with the fiber/fiber bonds resulting in a weaker, more inferior product. In order to maintain good edgewick sizing properties for lactic acid and citric acid, components of dairy products and fruit juices, high levels of alkyl ketene dimer (AKD) which is a cellulose reactive sizing agent, cationic thermosetting resins and retention aids are used to promote "hard" sizing along the edge of the board. Other sizing agents such as alkenyl succinic anhydride (ASA) have been tried but failed to adequately stop lactic acid penetration.

The present invention improves the edgewick sizing of liquid packaging board through the incorporation of calcium carbonate filler, preferably ground calcium carbonate or precipitated calcium carbonate (PCC) in amounts of up to 10 percent in the board. The calcium carbonate filler provides increased efficiency of the sizing agent and, moreover, alkyl ketene dimer (AKD) or alkenyl succinic anhydride (ASA), preferably AKD, can be utilized in these paperboard products in the presence of calcium carbonate filler to provide superior sizing properties.

It has also been surprisingly and unexpectedly discovered that the use of calcium carbonate as filler in paperboard used in liquid packaging results in a synergistic interaction between the calcium carbonate and a sizing agent selected from the group consisting of alkyl ketene dimer, alkeknyl succinic anhydride, cationic polyamidoamines, styrene maleic anhydride and mixtures thereof additive that further enhances edgewick sizing properties. This sizing agent provides the liquid container paperboard with greater wet strength durability, as measured by test method T456; OM-87, Technical Association of the Pulp and Paper Industry (TAPPI). When the precipitated calcium carbonate is first surface treated with either sodium stearate, an acrylic copolymer, or a salt of a C_{12} - C_{20} fatty acid, the

paperboard's edgewick sizing properties are further enhanced. Preferred acrylic copolymers comprise acrylonitrile copolymers consisting of butylacrylate, butylmethacrylate, 2-ethylhexylacrylate, methyl methacrylate, styrene, vinyl acrylate, vinyl chloride and mixtures thereof.

The incorporation of calcium carbonate as filler into liquid container paper board improves the retention of the alkyl ketene dimer size by up to twenty-eight percent (28%) and dimer reactivity by up to twenty-five percent (25%). This provides two benefits as this improves the board's edgewick sizing properties while the incorporation of the calcium carbonate results in increased paperboard bulk, a stabilized system alkalinity and subsequently reduces or eliminates entirely the need for additional sodium bicarbonate. This in turn also reduces the need to incorporate costly thermosetting resins in the paperstock as well.

The calcium carbonate filler further results in increased paperboard brightness, smoothness and better polyethylene or wax coating coverage. The paperboard possesses enhanced stiffness and rigidity as the calcium carbonate stabilizes the paperboard systems' alkalinity and acts to improve the systems retention of alkyl ketene dimer.

The manufacture and preparation of the liquid paperboard containing the calcium carbonate filler is much the same as that of ordinary paperboard. The wood pulp is beaten, refined and diluted to produce a fiber slurry or furnish. Calcium carbonate is added to the furnish separately or together with the other starches, resins and sizing agents. Whereas both ground calcium carbonate (GCC) as well as precipitated calcium carbonate (PCC) could be utilized as filler in the present invention, precipitated calcium carbonate (PCC) affords a superior paper product. Controlling the particle size, particle surface area, etc. enables the manufacturer to tailor the calcium carbonate to specific paperboard products.

The paperboard sheet is then formed on a paper machine whereby the water is screened out and the paper dried by passing it over a series of heated drums, The paper is

then calendered at high speed in order to impact the desired degree of smoothness. Liquid paperboard is generally coated on both sides with a polyethylene film using film forming applications known in the art. The thickness of the film will depend upon the liquid to be contained. Controlling factors include acidity and alkalinity, storage temperatures, exposure to sunlight, etc.

The calcium carbonate, can be incorporated into the liquid paperboard in amounts of from about 0.5 weight percent to about 10.0 weight percent based on the total weight of the paperboard. Preferably the amount of calcium carbonate is from about 3.0 weight percent to 10 weight percent and most preferably from about 4.0 weight percent to about 6.0 total weight percent. Other components comprise wet strength agents such as polyamine epichlorohydrin resins that further bind the pulp fibers together for increased paper strength and durability. Natural pitch resins also provide the same binding function. Retention aids such as anionic polyacrylamides also bind and stabilize the components of the system.

The following non-limiting examples are provided to more specifically teach and set forth particular embodiments of the present invention as envisioned here. They are for illustrative purposes only however, and it is recognized that minor changes and alterations can be made to the process parameters and components that are not specifically contemplated herein. However, to the extent any such changes or alterations do not materially change or effect the process or the final product, it is to be understood that such changes also fall within the spirit and scope of the invention as defined by the claims that follow.

EXAMPLE 1

PCC Filled Liquid Packaging Board to Improve Edgewick Sizing

PCC filler was used in liquid packaging board to determine its effect on edgewick sizing. Paperboard handsheets were made with Prince Albert bleached northern kraft pulp at a 75 percent hardwood/ 25 percent softwood blend. The hardwood and softwood pulps were separately refined at 1.6 percent consistency to an endpoint of 400 and 500 Canadian Standard Freeness respectively. The refined pulp was diluted to 1.0 percent consistency for papermaking. The target sheet weight was 225 grams per meters squared conditioned. The concentration of alkyl ketene dimer (AKD) size (Heron® 75; Hercules Inc.) was held constant and added to the furnish at 0.125 percent (2.5 pounds per ton) based on dry sheet weight. Cationic potato starch (Sta-Lok 400®; A.E. Staley Mfg. Co.) was added to the furnish at 0 percent, 0.5 percent, and 1.0 percent (0, 10, and 20 pounds per ton) based on dry sheet weight. A thermosetting resin, polyamide-amine epichlorohydrin (PAE) (Kymene® 557H; Hercules Inc.) was added to the furnish at 0 percent, 0.25 percent and 0.5 percent (0,5, and 10 pounds per ton). ALBACAR® LO PCC was added to the furnish in amounts adjusted to achieve levels of approximately 0 percent, 3 percent, and 6 percent. The amount of the anionic polyacrylamide retention aid (Accurac® 171; Cytec Industries) was held constant and added to the furnish at 0.00875 percent (0.175 pounds per ton) based on dry sheet weight. After the handsheets were formed on the Formax (Noble & Wood) sheet former, they were double-pressed between stainless steel rolls at 20 pounds per square inch. In the first pressing the sheet was still on the forming wire, sandwiched between pieces of papermachine "wet felt" material. The sheet was then removed from the forming wire, sandwiched between two pieces of unsized blotting paper, and pressed again. The handsheets were conditioned and tested under TAPPI standard conditions of 23 degrees Celcius and 50 percent relative humidity. Sheets were laminated, cut to size, and soaked for four hours in a 1 percent lactic acid solution for the edgewick testing. The results of the edgewick testing are shown in Table 1.

TABLE 1

EFFECT OF PCC FILLER ON EDGEWICK SIZING

Internal Starch (%)	PAE (%)	PCC Filler (%)	Edgewick Sizing (g/100 inches)
0	0	0.0	5.90
0	0	2.8	1.01
0	0	6.0	1.82
0.5	0	0.0	3.46
0.5	0	2.9	0.76
0.5	0	6.0	0.80
0	0.25	0.0	0.79
0	0.25	2.9	0.63
0	0.25	5.7	0.66
0.5	0.25	0.0	0.75
0.5	0.25	2.9	0.62
0.5	0.25	6.0	0.63
1.0	0.5	0.0	0.72
1.0	0.5	2.7	0.69
1.0	0.5	5.8	0.62

A review of the data shows that PCC filler improves the edgewick sizing of the liquid packaging board under all wet end conditions evaluated. The first set of data shows that board without starch, PAE, and PCC filler had an edgewick sizing value of 5.90 grams per 100 inches of exposed edge. This sizing value was significantly improved with the inclusion of PCC filler to the paperboard. Upon further review of the data, edgewick sizing values were always lower (better) when PCC filler was added to the paperboard whether starch, PAE, or both were present in the board as well.

EXAMPLE 2

PCC Filled Liquid Packaging Board to Improve Hot and Cold Beverage Containment

PCC filler was used in liquid container paperboard to determine the effect on hot and cold beverage containment. The handsheets were made with Prince Albert bleached northern kraft pulp at a 75 percent hardwood/25 percent softwood blend. The hardwood and softwood pulps were separately refined at 1.6 percent consistency to an endpoint of 400 and 500 Canadian Standard Freeness respectively. The pulps were blended in a 75:25 ratio. The refined pulp was diluted to 1.0 percent consistency of papermaking. Target sheet weight was 225 grams per meter squared conditioned. AKD size (Hercon® 75; Hercules Inc.) was held constant and added to the furnish at 0 percent, 0.5 percent, and 1.0 T percent (0,10, and 20 pounds per ton) based on dry sheet weight. PAE (Kymene® 557H; Hercules Inc.) was added to the furnish at 0.125 percent (2.5 pounds per ton) based on dry sheet weight. Cationic potato starch (Sta-Lok® 400 A. E. Staley Mfg. Co.) was added to the furnish at 0 percent, 0.25 percent, and 0.5 percent (0,5, and 10 pounds per ton) based on dry sheet weight. ALBACAR® LO PCC was added to the furnish in amounts adjusted to achieve levels of approximately 0 percent, 3 percent and 6 percent. Anionic anionic polyacrylamide retention aid, respectively. (Accurac® 171; Cytec Industries) was held constant and added to the furnish at 0.00875 percent (0.175 pounds per ton) based on dry sheet weight. After the handsheets were formed on a Formax (Noble & Wood) sheet former, they were double-pressed between stainless steel rolls at 20 pounds per square inch. In the first pressing the sheet was still on the forming wire, sandwiched between pieces of papermachine "wet felt" material . The sheet was then removed from the forming wire, sandwiched between two pieces of unsized blotting paper, and pressed again. The handsheets were conditioned and tested under TAPPI standard conditions of 23 degrees Celsius and 50 percent relative humidity.

Sheets were tested for Cobb sizing (3 minute test per TAPPI Test Method T441-OM90) using hot coffee (84 degrees Celsius., 8.4 grams per liter instant coffee) and cold

coffee (23 degrees Celsius, 8.4 grams per liter instant coffee). The results of the Cobb testing are shown in Table 2.

TABLE 2

EFFECT OF PCC FILLER ON COBB SIZING

Internal Starch (%)	PAE (%)	PCC Filler (%)	Hot Liquid Cobb Sizing (g/m^2)	Cold Liquid Cobb Sizing (g/m^2)
0	0	0.0	320	279
0	0	2.8	241	64
0	0	6.0	240	75
0.5	0	0.0	296	50
0.5	0	2.9	103	44
0.5	0	6.0	88	47
0	0.25	0.0	63	45
0	0.25	2.9	49	43
0	0.25	5.7	50	42
0.5	0.25	0.0	60	43
0.5	0.25	2.9	49	42
0.5	0.25	6.0	49	39
1.0	0.5	0.0	59	41
1.0	0.5	2.7	48	44
1.0	0.5	5.8	51	45

Results from this work show that PCC filler greatly improves the ability of the liquid containment board to hold hot and cold beverages as measured by hot and cold Cobb size testing. The first set of data shows that board without starch, PAE, and PCC filler had a hot liquid and cold liquid Cobb sizing value of 320 grams per meter squared and 279 grams per meter squared, respectively. This sizing value was significantly improved with the inclusion of PCC filler to the paperboard. Upon further review of the data, Cobb sizing values were always lower (better) when PCC filler was added to the paperboard whether starch, PAE, or both were present in the board as well.

EXAMPLE 3

Calcium Carbonate Filled Liquid Packaging Board to Improve edgewick Sizing

Ground calcium carbonate (GCC) and PCC filler were used in liquid packaging board to determine the effect on edgewicking sizing. This board was made using a pilot papermachine. Fillers were evaluated up to 10 percent in the liquid packaging board. Fiber furnish consisted of 80 percent bleached kraft hardwood and 20 percent bleached kraft softwood. Target board weight was 225 grams per meter squared. Cationic potato starch (Sta-Lok® 400; A. E. Staley Mfg. Co.) was added to the furnish at 10 pounds per ton. AKD size (Hercon® 79; Hercules Inc.,) was added at 0.2 percent and PAE (Kymene ® 557H; Hercules Inc.) was added at 10 pounds per ton to the thin stock. The calcium carbonate fillers were added to achieve levels in the sheet of 3 percent, 6 percent, and 9 percent or as close as possible. The filled board was conditioned and tested under TAPPI conditions of 23 degrees Celsius and 50 percent relative humidity. Sheets were laminated, cut to size, and soaked for four hours in a 1 percent lactic acid solution for the edgewick testing. The results of the edgewick testing are shown in Table 3.

TABLE 3

Effect of $CaCO_3$ Filler on Edgewick Sizing

Filler Type	Filler Level (%)	Edgewick Sizing (g/100 inches)
Unfilled Board	0.0	0.53
PCC - Calcite	3.7	0.49
PCC - Calcite	6.9	0.44
PCC - Calcite	9.6	0.39
PCC - Aragonite	3.2	0.54
PCC - Aragonite	6.2	0.46
PCC - Aragonite	9.6	0.46
GCC - Calcite	3.1	0.48
GCC - Calcite	7.1	0.38
GCC- Calcite	10.4	0.51

A review of the data shows that different mineral forms of calcium carbonate fillers will improve the edgewick sizing performance of liquid packaging board. Liquid packaging board without any filler present had an edgewick sizing value of 0.53 grams per 100 inches (g/100 in.) of exposed edge. Calcite PCC filler when added to the paperboard at levels up to 9.6 percent yielded edgewick values down to 0.39g/100 in. Calcite ground calcium carbonate (GCC) filler improved edgewick sizing down to 0.38g/100 in. at a filler level of 7.1 percent. However, at a level of 10.4 percent the sizing value increased to 0.51grams per 100 inches., still below the unfilled paperboard sizing value.

EXAMPLE 4

Use of PCC Filler and Surface Treated PCC Filler in AKD or ASA Sized Liquid Packaging Board to Improve Edgewick Sizing

The same procedure was followed as in Example 1 for making board handsheets using a Noble and Wood type sheet former. Hardwood and softwood pulps were blended in a 50:50 ratio. The target board weight was 225 grams per meter squared. Starch was added at 0.5 percent and cellulose reactive sizing agents (AKD or ASA) were added at 0.15 percent for AKD and 0.1 percent for ASA. PCC filler was added to achieve levels in the board of 3 percent, 6 percent, and 9 percent. The PCC filler can also be surface treated to improve edgewick sizing even more. For this example, the PCC filler was surface treated with 0.5 percent acrylic copolymer. The results of the edgewick sizing testing are shown in Table 4.

TABLE 4

Effect of Using PCC Filler on Edgewick Sizing of AKD or ASA Sized Liquid Packaging Board

Filler Type	Filler Treatment	Filler Level (%)	Sizing Agent	Edgewick Sizing (g/100 inches)
none	- -	0.0	AKD	1.62
PCC	none	4.8	AKD	0.92
PCC	none	9.3	AKD	1.10
PCC	0.5% acrylic copolymer	5.0	AKD	0.90
PCC	0.5% acrylic copolymer	9.1	AKD	0.83
none	- -	0.0	ASA	2.40
PCC	none	5.4	ASA	1.41
PCC	none	10.7	ASA	1.90
PCC	0.5% acrylic copolymer	5.2	ASA	1.20
PCC	0.5% acrylic Copolymer	9.3	ASA	1.18

A review of the data shows that PCC filler will improve the edgewick sizing performance of liquid packaging board made with either AKD as the internal sizing agent or less commonly used ASA as the internal sizing agent. It is also shown that by surface treating the PCC filler with an acrylic copolymer, the edgewick sizing performance is further enhanced. For paperboard sized with AKD containing no filler, an edgewick sizing value of 1.62 grams per 100 inches (g/100 in.) of exposed edge was achieved. The addition of PCC filler at levels of 4.8 percent and 9.3 percent improved edgewick sizing values to 0.92 g/100 in. and 1.10g/100 in., respectively. When the PCC filler was surfaced treated with 0.5 percent acrylic copolymer (dry weight on dry weight basis) and then added to the paperboard at levels of 5.0 percent and 9.1 percent, sizing values were further reduced to 0.90 g/100 in. and 0.83g/100in., respectively.

For paperboard sized with ASA containing no filler, an edgewick sizing value of 2.40 grams per 100 inches of exposed edge was achieved. The addition of PCC filler at levels of 5.4 percent and 10.7 percent improved edgewick sizing values to 1.41 and 1.90g/100in., respectively. When the PCC filler was surfaced treated with 0.5 percent acrylic copolymer (dry weight on dry weight basis) and then added to the paperboard at levels of 5.2 percent and 9.3 percent, sizing values were further reduced to 1.20 g/100 in. and 1.18g/100in., respectively.

What is claimed:

1) A liquid container paperboard comprising calcium carbonate wherein the said calcium carbonate is used in an effective amount to enhance edgewick sizing properties and reduce liquid penetration.

2.) The liquid container paperboard of claim 1 wherein the paperboard further comprises a sizing agent selected from the group consisting of alkyl ketene dimer, alkenyl succinic anhydride, cationic polyamidoamines, styrene maleic anhydride and mixtures thereof.

3.) The liquid container paperboard of claim 1 wherein the said calcium carbonate is selected from the group consisting of ground calcium carbonate and precipitated calcium carbonate.

4.) The liquid container paperboard of claim 3 wherein the said calcium carbonate is precipitated calcium carbonate.

5.) The liquid container paperboard of claim 4 wherein said precipitated calcium carbonate is coated with a surface coating selected from the group consisting of sodium stearate, acrylic copolymers, salts of C_{12} - C_{20} fatty acids and mixtures thereof.

6.) The liquid container paperboard of claim 5 wherein said acrylic copolymers comprises one or more monomers selected from the group consisting of butylacrylate, butylmethacrylate, 2-ethyl hexylacrylate, 2-hydroxyethlacrylate, methylmethacrylate, styrene, vinyl acrylate, vinyl chloride and mixtures thereof.

7.) The liquid container paperboard of claim 4 wherein said precipitated calcium carbonate is incorporated into said paperboard in an amount from about 0.5 weight

percent up to about 10.0 weight percent of the total weight of the container paperboard.

8.) The liquid container paperboard of claim 7 wherein said precipitated calcium carbonate is incorporated into said paperboard in an amount of from about 3.0 weight percent to about 10.0 weight percent.

9.) The liquid container paperboard of claim 8 further comprising a polyethylene film that coats the surface thereof.

10.) The liquid container paperboard of claim 9 further comprising wet strength agents, retention aids and optical brightening agents.

11.) A method for the reduction or elimination of edgewick sizing in liquid packaging paperboard comprising the incorporation of an effective amount of precipitated calcium carbonate as a filler.

12.) The method of claim 11 wherein said paperboard also comprises a sizing agent selected from the group consisting a alkyl ketene dimer, alkenyl succinic anhydride, cationic polyamidoamines, styrene maleic anhydride, cationic size resin and mixtures thereof.

13.) The method of claim 11 wherein said precipitated calcium carbonated is pre-treated with a surface coating comprising sodium stearate, acrylic copolymers, salts of C_{12} - C_{20} fatty acids and mixtures thereof.

14.) The method of claim 13 wherein said acrylic copolymers comprise one or more monomers selected from the group consisting of butylacrylate, butylmethacrylate, 2-ethylhexylacrylate, 2-hydroxyethlyacrylate, methylmethacrylate, styrene, vinyl acrylate, vinyl chloride and mixtures thereof.

15.) The method of claim 11 wherein said precipitated calcium carbonate is incorporated into said liquid packaging paperboard in an amount from about 0.5 weight percent to about 10.0 weight percent based on the total weight of the paperboard stock.

16.) The method of claim 15 wherein said precipitated calcium carbonate is incorporated into said liquid packaging paperboard in an amount of from about 3.0 weight percent to about 10 weight percent based on the total weight of the paperboard stock.

INTERNATIONAL SEARCH REPORT

	tional Application No PCT/US 99/31136

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 D21H27/10 D21H21/16 //D21H17:67

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 D21H

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	claims 1-3,6,9,11	5
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A	EP 0 406 932 A (ACEC UNION MINIERE) 9 January 1991 (1991-01-09) the whole document	11,15

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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20 July 2000	27/07/2000

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Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

Information on patent family members

In tional Application No
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